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Multi-scale mechanical analysis of thin titanium layer on UHMWPE substrates for biomedical applications

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Abstract

We present a multi-scale scratch resistance analysis of thin titanium layer with thickness varying between 0.5 and 1 µm, deposited on two biomedical polymeric substrates (UHMWPE). Using different experimental set-ups with specific tip radii, we have performed scratch experiments at various testing conditions. First, nano scratch tests have been performed with a spherical indenteur (tip radius R = 22µm) with increasing progressively the normal applied load between 1 and 100 mN. Then, macro-scratch tests have been realized with a spherical indenter (tip radius R = 200 µm) with the same experimental procedure by applying normal load in the range of 0.5 to 15 N. Finally, experimental results about multi-pass scratch tests at low average contact pressure are presented. Scratch experiments have been performed using a specific built-in microvisioscratch device that allows in situ observations of the contact area and of the residual groove during tests. About 100 repeated scratch experiments have been realized in the same track with a glass spherical indenter (tip radius R = 3.3 mm) at a constant normal load of 5 N. Friction coefficient evolutions are studied as function of the scratching conditions and some correlations have determined with damage mechanisms observed by SEM and chromatic confocal microscopy.

Keywords: titanium thin films, multiscale scratch test, sliding fatigue

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1. Introduction

The tribological performances of any coated surface depend on the application environment, the mechanical properties of the coating/substrate system, the adhesion between the coating and its substrate and also the fracture mechanisms. All these properties are directly related to the microstructure, density, composition, internal stress, defects or phase contents of the coating and of the near surface region of the substrate. For many applications, especially for biomedical applications (prosthetic implants), whatever the function of the coating, the main problem deals with the quantification of the mechanical resistance as a function of the coating deposition process and related parameters, but also the surface preparation of the underlying substrate. The main difficulty is then to estimate the lifetime in service of the coated system, according to simple mechanical tests. Classically, indentation and scratch tests are used in order to estimate respectively hardness and elastic modulus, but also critical load related fracture and/or adhesion failure [1-4]. A new aspect of current study, especially in the case of coated system, exhibiting high difference in mechanical behaviors between coating and its substrate, is the investigation of the fatigue behavior in the sliding wear using a modified scratch system [5-7]. In the present paper, we present the main results using an original multi-mode scratch tests procedure at different scales in order to reproduce progressively submicroscopic contacts to macroscopic contact. All tests have been conducted on a coated system, composed by a thin metallic titanium layer deposited on polymeric substrate.

2. Experimental details

Titanium thin films, with a thickness of 0.5 µm were obtained using a PVD deposition technique. Thin layers have been deposited on UHMWPE polymeric substrates, whose surfaces were prepared using mechanical polishing. Prior to deposition the substrates were placed in an ultrasonic in ethanol for 15 min and then dried in hot air to remove residual particles. Progressive load scratch test procedure has been conducted first using a Nano Scratch Tester (CSM Instruments) with a 20 µm tip radius diamond indenter and then using a Macro Revetest Scratch Tester (CSM instruments) with 200 µm tip radius Rockwell diamond indenter. At the nanoscale level, the diamond tip was drawn across the coating with a sliding speed of 600 $\mu m.min^{-1}$, corresponding to loading rate varying between 8 and 14 $mN.min^{-1}$, as a function of the normal load. For tests #1.1 and #1.2, normal loads were applied between 1 to 30 mN and 5 to 50 mN, respectively. At the microscopic and macroscopic levels, the sliding speed was imposed at 5 $mm.min^{-1}$, corresponding to loading in the range of 7 $N.min^{-1}$. In this case, normal loads during ramp tests were fixed between 1 to 15 N.

To assess the wear and fatigue resistance of the coated system, unidirectional multiple scratch tests in the same track, along the same scratch line (sliding in only one direction) were performed. Similar procedure has been used for adhesion study of TiN coatings [5, 6] and of ceramic layer [8]. By comparison with these different previous studies [5, 6, 8], we have developed a specific apparatus allowing in situ optical observations of the contact and of the residual wear track during each scratch test. To test non transparent materials, a spherical glass indenter with a tip radius of 3.3 mm was loaded at a constant normal applied load of 5 N and at a constant velocity of 0.1 $mm.s^{-1}$ over a scratch length of about 3 mm. Our built in system is detailed in Ref. [7, 9].

Using optical microscopy, but also scanning electronic microscopy, examinations at different magnification of the wear track were assessed in order to identify failure mechanisms during mechanical tests.

3. Results

Figure 1 shows for nanoscratch test the resistance to penetration curves, exhibiting the penetration depth as a function of the normal applied load, and also the evolution of the apparent friction coefficient as a function of the scratch distance. Whatever the normal load applied, the resistance to penetration curve is not really sensitive to the loading rate and the apparent friction coefficient appears to be constant over the scratch length. Oscillations observed in the different curves in Fig. 1 are related to the initial roughness of the coated systems and certainly to failure mechanisms during scratch experiments. Optical observations of the residual scratch groove show some adhesive failure but also microscopic brittle damage of the metallic coating. Such surprising damage mechanisms for a ductile metallic coating are due to the low stiffness of the underlying polymeric substrate that induces a rapid and important penetration of the rigid into the bilayer system. Different critical load may be identified using micrographs. They suggest that the soft substrate reaches a critical deformation for the metallic coating under the effects of the scratch indenter and the coating failure occurs. More accurate analysis and correlations with optical observation are needed to identify the different critical loads, corresponding to the transitions of mechanical behavior. However, as suggest in a previous paper [4], the critical load is not really an intrinsic parameter and some average physical parameters, such as the contact pressure, the average strain and the average strain rate, have to be estimated at each transition. Such analysis is in progress. Note that For biological applications in biomedical device, nanoscratch tests evidence that no wear or scratch particles were detached around the residual groove even at high load and high penetration depth with a ratio h_T/t rapidly greater than 10 (with h_T, the penetration depth and t, the film thickness).

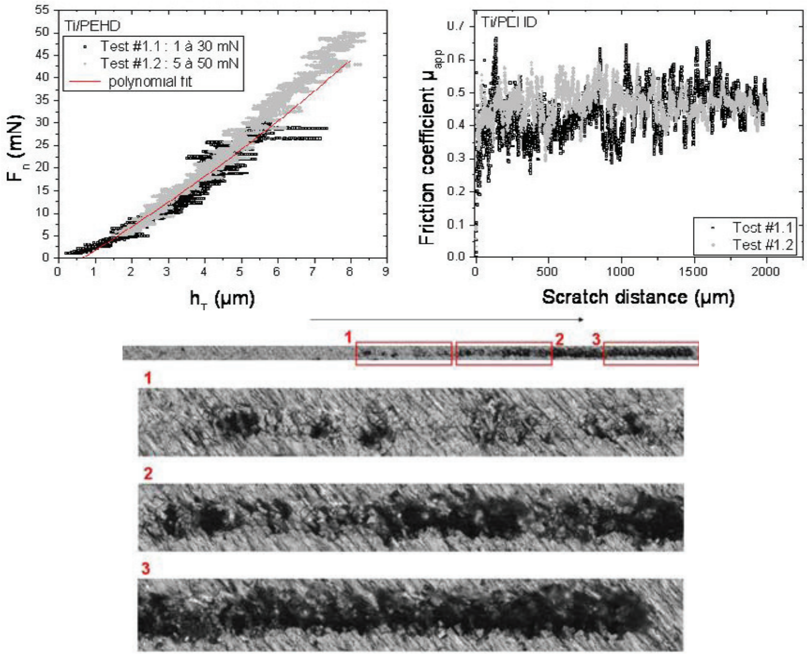

Fig. 1. (a) Resistance to penetration curve and (b) apparent friction coefficient with optical micrographs of the residual groove (test #1.1) obtained with the Nano Scratch Tester (CSM).

In figure 2, we have represented evolutions of the tangential load F_T, and the penetration depth as a function of the normal applied load F_N during macro-scratch experiments using a Macro Revetest tester (CSM). It is interesting to note that the h_T vs F_N curve is a polynomial function and the evolution of the tangential load is roughly linear without any oscillations. The apparent friction coefficient, defined by the ration F_T/F_N is constant along the scratch distance, with an average value of 0.32. The difference in apparent friction coefficient value between nano (Fig. 1) and macro-scratch tests is directly related to the difference in average contact pressure imposed during scratch tests. As previously observed for nano-scratch experiments, the penetration depth of the sliding Rockwell indenter tip with a radius R = 200 µm increases dramatically during progressive load scratch test procedure to reach a value h_T = 140 µm at F_N = 15 N; corresponding to a ratio h_T/t > 200. Optical observations (Fig. 2), confirmed by SEM observations (Fig. 3) describe local damage or failure events, located only in the residual groove. Although the penetration depth is very high, no delamination or micro adhesive chipping phenomenon, forming a specific network along the scratch edges, can be noted, indicating a good adhesion of the Ti coating on its polymeric substrate. Due to the high penetration depth by comparison with the film thickness, it is not obvious to observe some delamination in the bottom of the track.

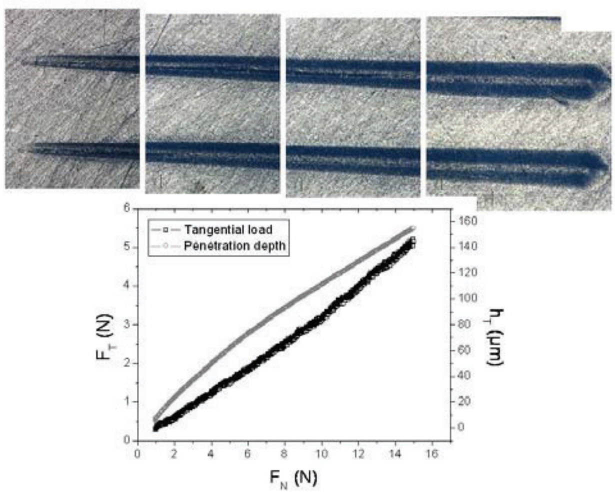

Fig. 2. Tangential load and penetration depth as a function of the normal applied load, using Macro Revetest (CSM) and corresponding optical micrographs of the residual groove.

To tend progressively to real macroscopic contact and then to reproduce solicitation in service, we have modified our built in scratch system [9] in order to investigate during repeated sliding the fatigue – wear of metallic thin coatings deposited on polymeric substrate. Extension of our Microvisio Scratch system [9] to give low load repeated passes over the same track, will allow detecting progressive damage at each pass, using both the friction force transducer [5], but also in situ optical observations. As observed in Fig. 4, at each pass, the apparent friction coefficient is monitored as a function of the sliding distance. In parallel, three pictures are also recorded at the beginning, the middle and at the end of scratch track. In figure 4, optical pictures recorded in the middle of the groove are presented for the different passes, with corresponding evolution of the apparent friction coefficient. Note that with our multi-pass testing, the moving indenter tip is in contact with the deformed surface, when pictures are recorded.

During the first passes (N < 10) the contact between the glass spherical indenter is mainly elastic, with a more or less symmetric contact. However, the friction coefficient increases slightly with passes, indicating a small adhesive phenomenon between the tip and the metallic surface, certainly due to the formation a very thin layer of titanium on the surface of the moving indenter tip.

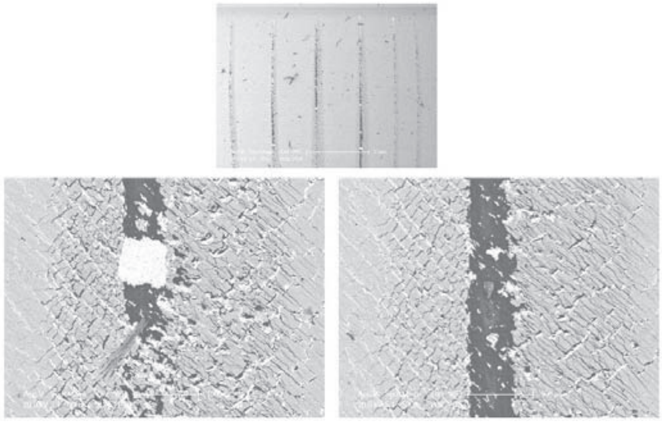

Fig. 3. SEM micrographs of the residual grooves obtained with the Macro Revetest Scratch Tester (CSM).

At pass N = 50, some micro-scratch tracks can be easily observed into the macro-groove formed during repeated tests, with local brittle failure of the Ti coating, as observed in Fig. 1 and then delamination as observed in Fig. 2 in the bottom of the scratch grooves. The apparent friction coefficient increases progressively as a function of the scratch distance. At pass N = 100, the friction is more constant along the scratch test and the average value is lower than value estimated at N = 50. In figure 4, optical micrograph shows for N = 100 that the number of the micro-scratch tracks in the macro-groove is dramatically increased and the Ti coating seems to partially removed in the track. This progressive delamination of the Ti coating is confirmed by SEM observations (Fig. 5) and important accumulation of titanium wear particles can be noted at the beginning and at the end of the macro-groove.

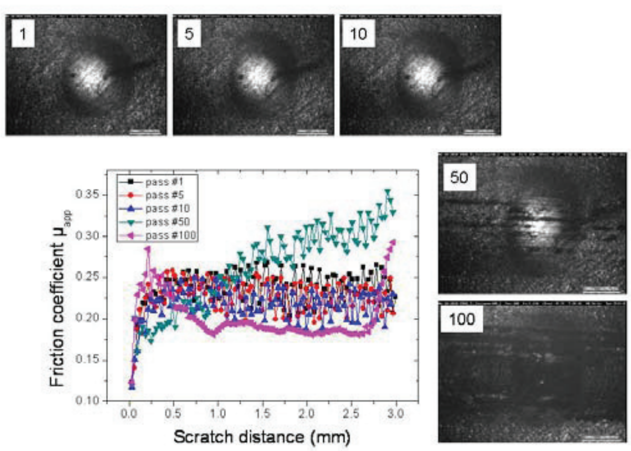

Fig. 4. Evolution of the apparent friction coefficient for different passes and corresponding in situ optical observations of the contact between the sliding tip and the deformed surface.

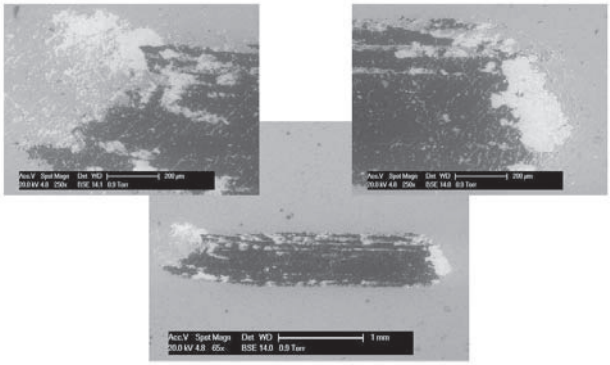

Fig. 5. SEM micrograph of the residual obtained after multiple scratch test procedure (after 100 passes).

4. Conclusions

The mechanical response of a very thin titanium coating deposited on a polymeric substrate has been studied in terms of multi-mode scratch tester. Nano and then macro-scratch experiments have demonstrated only local micro adhesive chipping phenomenon located in the residual groove that can be correlated to an excellent adhesion at the metallic coating polymeric substrate interface. Results about the sliding fatigue behavior by a modified scratch tester allowing in situ optical observations of the contact and the track have been presented. Interesting correlation can be found between failure mechanisms noted at the nanoscale level and those observed during macro-contacts.

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